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A STUDY OF VOLATILE CONTAMINANTS IN RECOVERED WATER

Technical Report No. 1

*Contract NAS 9-11580
SwRI Project 01-3096*

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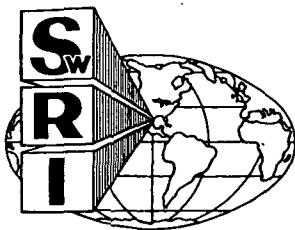
Prepared for

**Laboratory Support Section
Manned Spacecraft Center
National Aeronautics and Space Administration
Houston, Texas**

By

**Herbert C. McKee
Rudy Marek, Jr.**

May, 1972



**SOUTHWEST RESEARCH INSTITUTE
SAN ANTONIO**

HOUSTON

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SUMMARY AND CONCLUSIONS

This investigation was conducted to obtain data on the volatile organic impurities that occur in urine samples and in water recovered from urine by distillation. Additional tests were also carried out to determine the fate of iodine preparations used to prevent bacterial action.

A laboratory distillation apparatus was constructed which could be used for tests at either atmospheric pressure or reduced pressures. Tests were conducted at two pressures (atmospheric and 36 mm Hg), using urine samples and water samples containing "iodophor" premix formulations of commercial iodine-containing bactericides.

The results are summarized as follows:

- Iodine distilled out of the original material readily, and tended to concentrate in the first portions of distillate collected. Some iodine also disappeared by reaction with urine constituents over a period of time, and some also was lost during distillation and was presumed to have escaped to the atmosphere.
- Trace organic constituents present in urine were removed readily by distillation, and were concentrated in the first portions of distillate recovered. For nearly all constituents, concentrations in the first portion of distillate were significantly higher than in the original charge to the distillation unit.
- Distillation at reduced pressure reduced the amount of volatile impurities in the distillate, compared to results obtained at atmospheric pressure. This effect was presumed to result from an increased rate of thermal decomposition at the higher temperatures required for atmospheric distillation.

TABLE OF CONTENTS

<u>Section</u>		<u>Page</u>
I.	INTRODUCTION	1
II.	DESIGN OF LABORATORY DISTILLATION APPARATUS	2
III.	IODINE DISTILLATION TESTS	5
IV.	ANALYTICAL PROCEDURES	10
V.	DISTILLATION TEST PROCEDURE AND RESULTS	13
	A. Experimental Procedure	13
	B. Results and Discussion	14
APPENDIX		
Original Data Used in Preparing Table II		

ILLUSTRATIONS AND TABLES

	<u>Page</u>
<i>Figure 1. Diagram of Laboratory Distillation Apparatus</i>	3
<i>Table I. Results of Iodine Distillation Tests</i>	8
<i>Figure 2. Typical Chromatographic Chart Record</i>	12
<i>Table II. Summary of Distillation Data</i>	15

I. INTRODUCTION

In future space flights of long duration, water reclamation and reuse is contemplated as an integral part of the environmental control systems. As missions extend to longer and longer duration, problems of recovery and reuse will assume added importance.

Three types of contamination must be considered in planning methods for recovery and reuse of water, including (1) microbial contamination; (2) non-volatile chemical impurities; and (3) volatile materials. The problem of contamination by bacteria and other micro-organisms can be solved through sterilization or the use of antibiotics or other bactericides, although some additional consideration may be needed to insure freedom from viral contaminants. The problem of non-volatile constituents (largely inorganic salts) can be solved by proper design of distillation units or other equipment used in water recovery, since these usually will not be present in the recovered water to any extent if entrainment can be avoided. For recovery methods other than distillation such as reverse osmosis, proper design and operation can also be effective in separating most inorganic salts.

The problem of trace volatile constituents is somewhat more difficult, since vaporization and other methods of water purification may not separate volatile materials. In fact, some previous information indicated that volatile constituents could actually be concentrated by the distillation process and thus could occur in the recovered water in higher concentrations than those originally present. In addition, regardless of the method used for water recovery, contact with the atmosphere in the space cabin will also result in contamination by contact with trace atmospheric impurities during or after recovery.

This investigation was undertaken in order to obtain basic information on the volatile impurities present in urine samples and in water recovered from urine by distillation. The work reported here included laboratory distillation tests to determine the nature and extent of the volatile constituents in the distillate, and to evaluate possible problems in distillation due to iodine used for control of microbial contamination. Subsequent phases are also planned to develop design criteria for distillation equipment to minimize the problems of volatile contaminants, and to evaluate various methods which might be used for purification subsequent to recovery.

II. DESIGN OF LABORATORY DISTILLATION APPARATUS

To conduct laboratory distillation tests, apparatus was needed that could duplicate some of the operating conditions of actual space flight hardware, although duplication of all operating characteristics was obviously impossible since this would involve operation in a weightless environment. However, pressure, temperature, and distillation rate can be controlled over a wide range through the use of conventional laboratory apparatus. It was decided to conduct experiments at atmospheric pressure and also at 36 mm pressure, the latter figure being a design figure for possible space flight hardware. It was also necessary to collect separate fractions of distillate while a run was in progress in order to determine which portions of a distillation run resulted in the highest levels of volatile contaminants in the recovered water.

After evaluating several types and sizes of stills and other components, the apparatus shown in Figure 1 was assembled and tested for use in subsequent laboratory experiments. Major portions of the apparatus include the following:

1. Distillation flask. This is a 3-neck 1000-ml boiling flask heated by an electric heating mantle controlled by a variable transformer. Heat input can be varied over a wide range by variations of voltage, to produce the desired distillation rate.
2. Column. A 200-mm Vigreux distilling column was used to help prevent carryover of liquid droplets in the vapor stream.
3. Condenser. A West type condenser with a minimum length of 400 mm was used, and was cooled with ice water.
4. Receiver. A revolving type multiple distilling receiver was used which could be rotated to collect distillate progressively into any of four separate 50-ml flasks.
5. Vacuum system. The outlet of the receiver was fitted with a cold trap, a control valve, and a vacuum pump to control the vacuum at any desired value. The outlet of the cold trap was vented to the atmosphere for tests conducted at atmospheric pressure. The sample

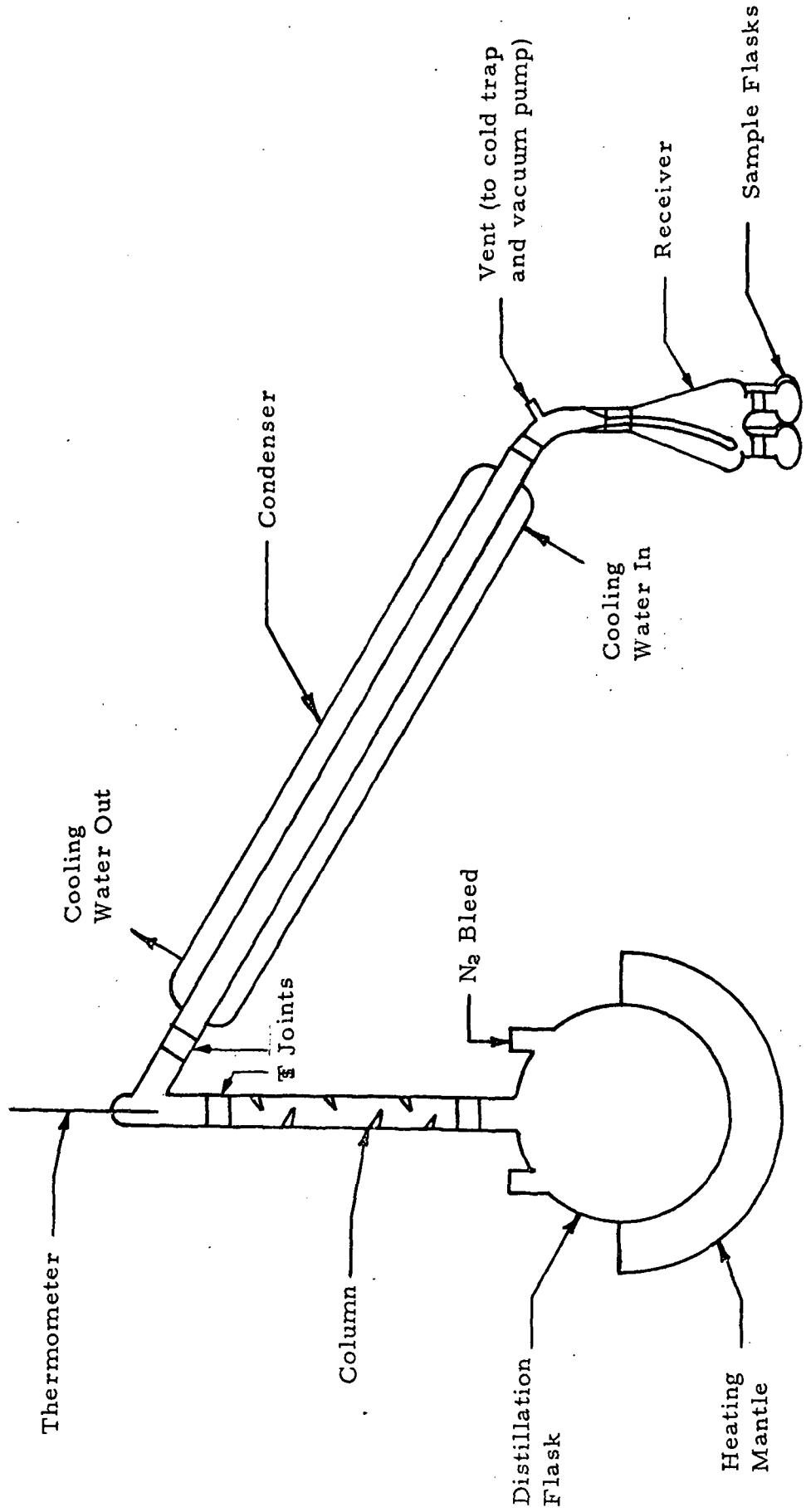


Figure 1. Diagram of Laboratory Distillation Apparatus

flasks were also cooled with ice water to prevent loss of volatile constituents after collection.

This assembly proved to be quite versatile in that pressure, temperature, and distillation rate could be controlled throughout the desired range. In addition, it was found helpful to bubble nitrogen through the sample to aid in distillation and to prevent uneven flashing, especially when distillation was conducted at reduced pressure.

III. IODINE DISTILLATION TESTS

Bacterial action occurs readily in most waste water, including the various sources that can be recovered for reuse during space flight. To prevent this, some method of sterilization is needed for both waste water and recovered water being stored for reuse. In urine distillation processes, present plans call for the use of an iodine-containing preparation prior to distillation. Previous experimental work has utilized organic iodine compounds available commercially as so-called "iodophor" preparations.

The word "iodophor" means iodine carrier, and is usually applied to an organic compound which increases the solubility of iodine and tends to stabilize the iodine in aqueous systems. Frequently, non-ionic surfactants are also added to give better germicidal activity. A number of commercial preparations are available including the following:

Biopal VRO-20	(General Aniline and Film Corp.)
PVP-Iodine (polyvinyl-pyrrolidone)	(General Aniline and Film Corp.)
Betadine (PVP)	(Purdue Frederick Company)
IX-91	(Oxford Chemical Company)
Prepodyne	(West Chemical Company)
Wescodyne	(West Chemical Company)
Rapidyne	(West Chemical Company)
Weladyne	(West Chemical Company)
Triodine	(West Chemical Company)

Present plans for water recovery during space flight call for the addition of a premix including an iodophor material and other constituents when urine is first collected for storage, in order to control micro-organisms, ammonia, and foaming during distillation. The following premix compositions have been used:

Premix No. 1

Iodophor	67.0%
Sulfuric Acid	9.4%
Antifoam AF (Dow Corning)	3.6%
Water	<u>20.0%</u>
	100.0%

Premix No. 2

Iodophor (Biopal VRO-20)	49.0%
Sulfuric Acid	6.8%
Antifoam AF (Dow Corning)	2.1%
Water	<u>42.1%</u>
	100.0%

No. 1 is usually added at the rate of one part premix to 99 parts urine, while No. 2 is added at the rate of 2.25 g of premix to 390 ml of urine.

Since iodine is volatile, the question arose concerning the possible fate of iodine in a distillation process and also concerning the possible effect that iodine distillation might have on the occurrence of other organic volatile constituents. Therefore, a series of experiments was undertaken to determine the extent to which iodine might be distilled over in a typical distillation apparatus. Runs were made at both 36 mm and atmospheric pressure using demineralized water and different iodophor formulations. Available iodine was measured by titration with sodium thiosulfate, in original samples, distillate fractions, residue, cold trap samples, and solutions used to wash out the condenser after each run.

In the first runs completed, substantial loss of iodine occurred, due apparently to volatilization and loss in the apparatus or out the vents to the atmosphere. This was reduced by using ice water to cool the condenser and by using a cold trap cooled with ice water in the vent line. In addition, the entire apparatus was washed following the run to remove iodine deposited on the walls, mainly on the inner surface of the condenser. By including the iodine recovered in the cold trap and that obtained from the condenser, a total recovery in excess of 70 percent was obtained in most runs.

Table I shows a tabulation of data from some of the more pertinent experiments that were conducted after reasonably good recovery was achieved. While minor quantitative differences occurred as the result of premix formulation and distillation characteristics, several consistent trends are evident. The most important is the volatility of the iodine-containing constituents, as shown by the low free iodine content of the residue. In all cases, a major portion of the iodine was carried over by the distillation process and was distributed between the distillate fractions, the cold trap, and that deposited on the walls of the condenser. If the condenser was not cooled with ice water, losses were excessive, and it is presumed that a major portion of the iodine not accounted for in these runs escaped to the atmosphere.

After loss on the condenser, the most important effect was the occurrence of iodine in the first fraction of distillate collected, with successively smaller portions occurring in subsequent fractions. Thus, it is evident that a major portion of the iodine would occur in the recovered water, with additional iodine passing into the atmosphere. The distribution between these two effects would depend on condenser temperature and the design and operating characteristics of the system.

In these tests, pressure appeared to be relatively unimportant. Tests conducted at 36 mm and 760 mm pressure showed essentially the same pattern. Also, the type of iodophor used had only a minor effect on the relative amounts of iodine in the distillate and condenser; the same general pattern was shown with all types tested.

In an actual water recovery unit, the loss of iodine from the still would result in a loss of microbiological activity in the remaining residue. This might not be a problem if subsequent charges of feed containing additional free iodine were added periodically, but should be kept in mind in the design and operation of space flight hardware if problems arise. The ultimate fate of iodine passing into the atmosphere also is not known; presumably, much of this would deposit on available surfaces and be removed from the atmosphere.

In addition to the distillation tests, a question was raised concerning the possible destruction of iodine by other constituents present in urine samples prior to distillation. Therefore, several samples of urine were treated with the prescribed amounts of iodophor premix. Within a short time (30-45 min), all of the available iodine was consumed by reaction with constituents of the urine.

Table I. Results of Iodine Distillation Tests

Iodophor Type	Pressure mm Hg	Start	1st 50 ml	2nd 50 ml	3rd 50 ml	4th 50 ml	Residue	Cold Trap	Condenser Walls	Total Recovered, mg	Percent Recovery
Betadine	36	50.8	5.4	5.4	3.8	2.5	1.3	-	17.8	36.2	71
Betadine	36	70.0	18.0	4.0	1.0	0	1.0	2.0	20.0	46.0	66
IX-91	36	110.5	40.6	8.8	3.0	1.0	1.0	-	30.0	84.0	76
- Wescodyne	760	98.8	40.0	3.0	1.0	1.0	0	22.0	3.0	70.0	70
Wescodyne	36	99.0	35.5	3.2	1.0	0	0	-	34.6	74.3	75
Rapidyne	36	119.0	42.4	5.7	2.0	1.0	1.0	-	39.0	91.1	77
Weladyne	36	95.5	34.0	7.0	4.0	1.0	1.0	-	34.0	81.0	85
Triiodine	36	105.0	37.0	4.0	4.0	1.0	0	-	30.0	76.0	73
PVP Iodine	36	50.0	19.3	0	0	0	0	4.0	18.0	41.3	83
PVP Iodine	760	50.8	19.0	0	0	0	0	5.0	21.0	45.0	88
Biopal VRO-20	36	216.0	26.0	5.1	0	0	5.2	25.4	71.0	132.7	62
Biopal VRO-20	36	230.0	28.0	7.6	1.3	0	6.5	19.0	115.0	177.4	77

Note: Figures given (except Pressure and Percent Recovery) are milligrams iodine in total sample. Original material in distillation flask at start of run was 500 ml.

To examine this question further, higher concentrations of premix were used in an attempt to determine what would be required if it was necessary to maintain free iodine for an extended period of time. For example, with a concentration of 1440 mg of available iodine per liter, it was found that the concentration was reduced to 152 mg per liter by 6 hr of stirring, to 127 mg per liter after 24 hr, and to 114 mg per liter after 48 hr. After 96 hr, no available iodine was detected. This might lead to problems if subsequent addition of wastes were made to a distillation apparatus containing a large amount of residue which had previously been depleted of iodine. Whether or not problems actually occurred would depend on whether or not the iodine present would be effective in killing all available bacteria before the iodine itself had been either reacted or distilled out of the residue.

Tests with distillate indicated that iodine which distilled over was collected readily by contacting the distillate with activated carbon. Therefore, in the actual case of iodophor preparations in space flight hardware, iodine in the recovered water likely would not be a problem unless the absorptive capacity of the carbon for iodine were exceeded.

The presence or absence of iodine did not appear to influence the occurrence of other volatile organic constituents which were measured in subsequent experiments.

IV. ANALYTICAL PROCEDURES

Organic constituents of aqueous samples can be measured in many ways. However, for most purposes, gas chromatography is the preferred method because of the selectivity and sensitivity of this method. In many applications, many individual compounds can be measured in a single sample, at concentrations in the parts per million or parts per billion range. Therefore, gas chromatography was the method of choice for this investigation.

A persistent problem in trace analysis is the processing of samples prior to analysis. Usually, the trace organic compounds must be separated from the water or other substrate in which they occur, and must be collected relatively free from water and other major constituents in a form that can be passed into the chromatographic instrument. Many techniques to accomplish this have been used in the past, such as stripping with helium or other inert gas, preliminary chromatographic separation to remove water and carbon dioxide, distillation of low-boiling compounds, extraction with an organic solvent, flash vaporization, and various combinations of these and other methods.

The previous experience of Southwest Research Institute in using these techniques was reviewed in order to select a procedure appropriate for this study. Based on this review, it was decided to use a method based on the analysis of air samples taken from an enclosed flask containing a liquid sample. This so-called "head space gas analysis" method has been used in the past to study food flavor constituents in aqueous samples and for other purposes. The "head space gas" is considered to be the air in a sealed container above a measured volume of liquid. Trace volatiles in the liquid can be separated by "salting out" with sodium sulfate, so that the head space gas will contain a substantial portion of the total volatile organic material. By sampling this gas, samples relatively free of water can be collected for analysis, thus avoiding major interference from water in the analytical step. The small amount of water vapor contained in the sample can normally be tolerated by most suitable column materials and by flame ionization detectors with no difficulty.

To analyze samples, a 5-ml portion was placed in a 10-ml serum vial containing 5 g of anhydrous sodium sulfate. (The flask was previously purged with dry nitrogen.) A rubber septum was placed over the vial, and the entire sample was then agitated in a mechanical shaker for 5 min to saturate the sample with sodium sulfate. The vial was then placed in a

water bath maintained at 80° C for 10 min, so that a major portion of the volatile constituents would be driven into the air space (head space) of the vial.

Samples of this gas were then obtained by inserting a 1-ml syringe through the rubber septum, filling and evacuating the syringe three times, and then removing it. After adjusting the volume to exactly 1 ml, the gas sample was then injected into the gas chromatograph.

Samples were analyzed with a Perkin-Elmer Model 900 gas chromatograph equipped with a flame ionization detector. Other detectors could be used, but the flame ionization detection is preferred because of lack of sensitivity to water vapor and also because of the inherent sensitivity of this detector. Various column materials were evaluated, based on the previous experience of Southwest Research Institute, to select a column suitable for this study. The column selected was a 3-ft-long x 1/8-in.-diameter copper column packed with Porapak Q. This column has been shown to give good baseline stability with highly sensitive detectors over a wide temperature range, and has given excellent results in analyzing urine samples in previous studies.

Figure 2 shows a typical chromatographic chart record obtained in the analysis of a distillate sample produced by urine distillation. Quantitative results can be obtained by comparing either peak height or peak area with calibration runs made with known amounts of the same compound. Unknown constituents were evaluated by comparing peak areas to obtain relative concentrations, since absolute values could not be obtained.

These methods were used in analyzing original samples, distillate fractions, and residue in the various distillation tests that were conducted.

Note: Numbers identify individual peaks
as shown in Table II.

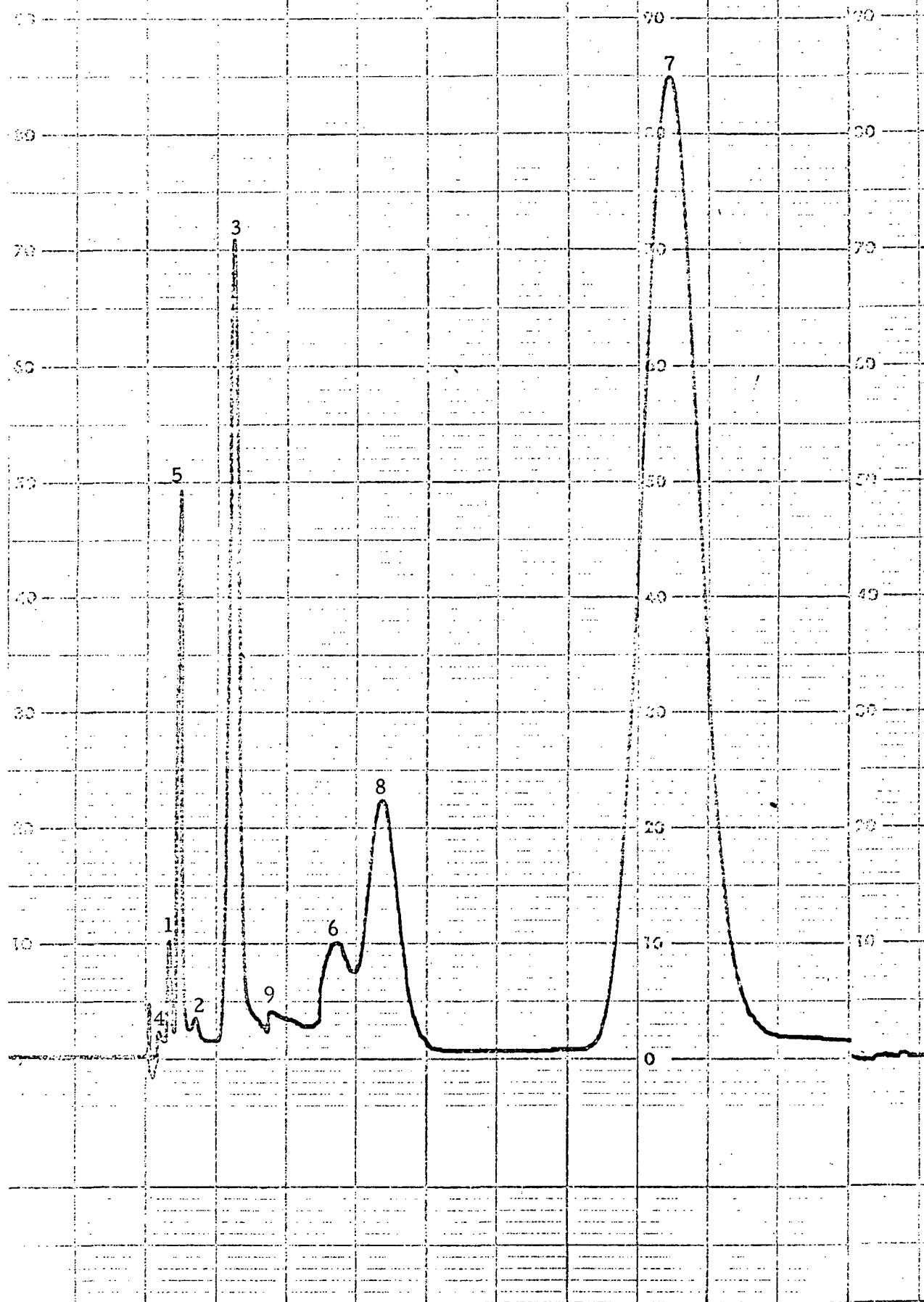


Figure 2. Typical Chromatographic Chart Record

V. DISTILLATION TEST PROCEDURE AND RESULTS

A number of distillation tests were completed, using different pressures and varying other operating variables. Complete tables of data from pertinent tests are included in the Appendix of this report to document the results obtained. The most important of these will be discussed here, and the procedures used will be outlined.

A. Experimental Procedure

All glassware used for sample storage, handling, and distillation was washed in chromic acid cleaning solution, rinsed with demineralized water, heated in an oven at 110° C for 4 hr, and purged with dry nitrogen prior to use. This step was found to be absolutely necessary to avoid contamination of samples and glassware by trace organic materials found in laboratory air.

Urine was collected in a 1-liter bottle fitted with a rubber stopper covered with metal foil, and was kept refrigerated at 2-3° C. The sample was treated with Biopal VRO-20 premix at the rate of 2.25 g premix per 390 ml urine. Distillation tests were conducted with a minimum of 500 ml of sample, which was placed in the boiling flask of the distillation apparatus.

Distillation was conducted by applying heat and making proper adjustments to maintain the desired vacuum, for tests at reduced pressure. Tests were conducted at atmospheric pressure by leaving the vacuum vent line open and adjusting heating rate as required. With either procedure, distillation could proceed at a reasonably uniform rate, and distillate was collected in the various receiving flasks in turn. Collection of the first 50-ml sample required approximately 25 min for vacuum distillation and 15 min for atmospheric distillation. After this collection, the receiving flask was then rotated to the next flask to collect a second distillation fraction, and so on in turn for a total of four fractions. After heating was discontinued, these four fractions were placed in small jars with screw caps and metal liners, together with 50-ml samples of the original sample, residue from the boiling flask, and any condensate from the cold trap. These samples were then placed in the freezer and kept frozen until they were analyzed. Immediately prior to analysis, they were thawed in order to remove portions for analysis as discussed previously.

B. Results and Discussion

Based on previous work with the same chromatographic column, three of the peaks were tentatively identified as methanol, ethanol, and acetone, on the basis of retention time. The acetone identification seems certain since acetone is a known constituent of urine and other body fluids; the other identifications are only tentative at this time. However, lack of positive identification does not prevent using the data to reach several conclusions concerning the behavior of volatile constituents during distillation.

On the basis of the tentative identifications, solutions containing 1 μ l per liter of methanol, ethanol, and acetone in water were prepared. Samples were analyzed by the head space technique to obtain data on elution times, together with positions and peak heights on the chromatographic chart. This then made it possible to convert the results of all runs to quantitative values, as shown in the table in the Appendix. A composite of the three solutions was also analyzed to be sure that no reaction among the three would occur that might change their positions and peak heights on the chart record.

The results of the final series of distillation runs are summarized in Table II. A total of nine constituents could be measured, using samples obtained by distillation at atmospheric pressure and at a pressure of 36 mm. As noted, relative concentrations in the various samples is expressed as a ratio compared to the concentration in the original charge used. That is, a value of 3.0 means that a given constituent occurred at three times the concentration of the original charge, while a value of 0.5 means that the constituent occurred at half of the original concentration.

Two of the volatile constituents occurred at low levels near the limit of detection (Nos. 6 and 9), and therefore significant quantitative results could not be calculated. As a rough approximation, these two constituents followed the same general pattern that occurred with other constituents that could be measured quantitatively.

Several consistent trends are obvious in examining Table II. The most obvious is the tendency for all of the constituents measured to concentrate in the first fractions of distillate collected near the beginning of a run. At atmospheric pressure, for example, the first fraction contained approximately eight times the concentration of the original charge for five of the seven constituents that could be evaluated quantitatively, with the other two ratios being 21.3 and 2.0. After the first fraction was collected, there was then a consistent tendency for concentrations

Table II. Summary of Distillation Data

<u>Atmospheric Pressure</u>	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)
Original Charge	1.0	1.0	1.0	1.0	1.0	0	1.0	1.0	P
1st Fraction	8.0	8.3	8.4	2.0	21.3	+	8.3	8.0	+
2nd Fraction	1.3	0.5	0.78	1.3	8.1	+	0.94	2.3	+
3rd Fraction	0.75	P	0.7	0.87	5.3	0	0.13	2.0	P
4th Fraction	0.37	P	0.48	0.75	3.8	0	0.02	1.0	P
Cold Trap	1.3	0	0.43	0	0	0	0	0	P
Residue	2.8	0	0.07	0.13	0.28	0	0	0	P
<u>Vacuum (36 mm)</u>									
Original Charge	1.0	1.0	1.0	1.0	1.0	0	1.0	1.0	1.0
1st Fraction	3.0	1.0	3.2	1.9	1.6	0	8.0	0.5	0
2nd Fraction	1.5	0	0.03	1.0	0.77	0	3.0	0	P
3rd Fraction	0.63	0	0	0.62	0.35	0	1.4	0	0
4th Fraction	0.38	0	0.03	1.2	0.18	0	0.71	0	0
Cold Trap	0.94	0	6.3	1.2	0.06	0	0.08	0	0
Residue	2.1	P	0.11	0.12	1.1	0	0	0.5	+

Notes:

- (1) Concentration of each constituent in original charge was arbitrarily assigned a value of 1.0. Other concentrations are expressed as a ratio relative to the original charge (to two significant figures).
- (2) P - present, but concentration too low to obtain quantitative results.
+ - present in sample, but ratio could not be calculated because concentration in original charge was zero.
- (3) Tentative identifications (based only on elution time):
 - (1) Methanol
 - (2) Ethanol
 - (3) Acetone

The remaining peaks were not identified.

to decrease successively in the second, third, and fourth fractions.

Another consistent trend was noted in the effect of pressure. With only one exception (No. 7), all constituents showed lower concentrations in the first fraction at 36 mm than at atmospheric pressure. Again, the second, third, and fourth fractions showed lower concentrations than the first fraction, confirming the tendency for the greatest amounts to distill over near the beginning of a run. The reason for the lower concentrations during vacuum distillation is not known, but likely is due to the effect of lower distillation temperatures in reducing thermal decomposition of higher molecular weight organic constituents. The occurrence of higher concentrations of some constituents in the residue, compared with the original charge, also indicates thermal decomposition during distillation.

Results were less consistent with respect to the levels of various trace organics found in the cold trap. Many constituents (Nos. 2, 5, 7, and 8, for example) were essentially absent, indicating either almost complete recovery ahead of the cold trap or else loss through the cold trap to the atmosphere. In an actual closed system, material collected in the cold trap in these experiments might escape to the atmosphere, depending on temperature and other operating variables. In at least one case (No. 3, vacuum), the cold trap contained a higher concentration than any of the other samples, due likely to vaporization out of the collected water samples at a pressure of 36 mm.

Concentrations in the residue after distillation were usually quite low compared to the original charge, indicating removal of a high percentage of each constituent during distillation. One notable exception occurred with No. 1; the higher concentration in the residue may have been due to thermal decomposition in this case.

While the data summarized here only include a limited number of individual compounds, the consistent trends likely apply to many other compounds that could not be measured with the analytical system used. However, it can be concluded as a general principle that low molecular weight organics will tend to concentrate in the first fraction of distillate collected to a considerable extent, and that the problem of volatile organic constituents is likely to be less serious if distillation can be conducted at reduced pressure, compared to the results obtained at atmospheric pressure.

APPENDIX

DATA SHEET OF CHROMATOGRAPHIC ANALYSIS OF URINE DISTILLATES

Compound	Peak No.	Distillation Pressure mm Hg	Standard		Urine Sample (500 ml)		1st 50 ml Distillate		2nd 50 ml Distillate		3rd 50 ml Distillate		4th 50 ml Distillate		Cold Trap		Residue (300 ml)	
			Peak* Areas	μg/100 ml	Peak* Areas	μg/100 ml	Peak* Areas	μg/100 ml	Peak* Areas	μg/100 ml	Peak* Areas	μg/100 ml	Peak* Areas	μg/100 ml	Peak* Areas	μg/100 ml	Peak* Areas	μg/100 ml
Methanol	1	760	16	80	16	80	128	640	20	100	12	60	6	30	20	100	44	220
Methanol		36	16	80	16	80	54	260	24	120	10	50	6	30	15	75	34	170
Ethanol	2	760	52	80	4	6	32	50	2	3	P	P	P	P	0	0	0	0
Ethanol		36	52	80	2	3	2	3	0	0	0	0	0	0	0	0	P	P
Acetone	3	760	464	80	268	46	2240	386	210	36	180	32	132	22	115	20	16	3
Acetone		36	464	80	212	36	680	116	6	1	1	0	4	1	1300	225	20	4
Unknown	4	760			16		32		20		14		12		0		2	
Unknown		36			16		30		16		10		20		20		2	
Unknown	5	760			36		768		292		190		136		0		10	
Unknown		36			34		56		26		12		6		2		36	
Unknown	6	760			0		50		20		0		0		0		0	
Unknown		36			0		0		0		0		0		0		0	
Unknown	7	760			3600		30,000		3400		450		60		0		0	
Unknown		36			1200		9600		3600		1650		850		100		0	
Unknown	8	760			30		240		70		60		30		0		0	
Unknown		36			20		10		0		0		0		0		10	
Unknown	9	760			P		32		8		P		P		0		24	
Unknown		36			P		0		P		0		0		0		8	

Note: P - present, but concentration too low for quantitative results.

* - chromatogram peak areas - arbitrary units of area.